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Synthesis of subphthalocyanine derivatives and their characterization

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Abstract

Subphthalocyanine and its derivatives which possessed substituents such as thiobutyl and thiophenyl were synthesized. These compounds were measured using spectroscopic measurements and cyclic voltammetry. Sensitization properties of synthetic subphthalocyanine (SubPC) in 2,4,6-Tris(trichloromethyl)-1,3,5-triazine as radical generation reaction in a poly(methyl methacrylate) film have been investigated by laser flash photolysis. © 2002 Elsevier Science Ltd. All rights reserved.

Keywords: Subphthalocyanine derivatives; Synthesis; Cyclic voltammetry; Laser flash photolysis

1. Introduction

Metal phthalocyanine (M-PC) derivatives and their related compounds have been of interest in molecular electronics, optoelectronics, photonics, etc. [1–5].

In general, M-PC derivatives have four isoindole units and a central metal. Some of the M-PC related compounds, subphthalocyanine (SubPC) derivatives are the lowest homologues consisting of three isoindole units and boron as the center (Fig. 1). Unsubstituted M-PC derivatives are known to have poor solubility in common organic solvents, whereas SubPC derivatives have excellent solubility since these phenomena are attributed to their non-planer crystal structure [6].

SubPC derivatives are only used as reagents for the ring enlargement reaction to prepare unsymmetry PC derivatives. Now, another application of SubPC derivatives has been reported [7,8]. In order to develop a new application of SubPC, we synthesized SubPC and its four derivatives such as hexakis(*S*-butyl)hexafyluoro-subphthalocyanine (SubPC(S-butyl)$_6$F$_6$), hexakis(*S*-phenyl)hexafyluoro-subphthalocyanine (SubPC(S-phenyl)$_6$F$_6$, dodecylkis(*S*-butyl)subphthalocyanine (SubPC(S-butyl)$_{12}$)

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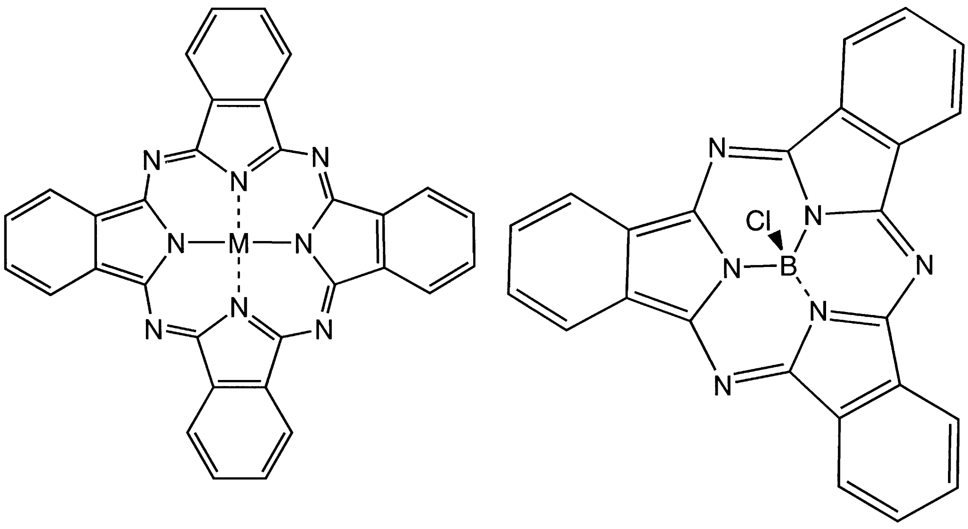


Metal phthalocyanine Subphthalocyanine

Fig. 1. Molecular structures of phthalocyanine and subphthalocyanine.

and dodecylkis(*S*-phenyl)subphthalocyanine (SubPC(*S*-phenyl)$_{12}$) (Scheme 1). We also synthesized Tris(*tert*-butyl)subphthalocyanine (*tert*- butyl-SubPC) and dodecylfluoro-subphthalocyanine (SubPCF$_{12}$) as standard compounds.

We report electrochemical properties and photochemical characteristics of SubPC in a photopolymer coating layer.

2. Experimental

2.1. Synthesis

2.1.1. Subphthalocyanine

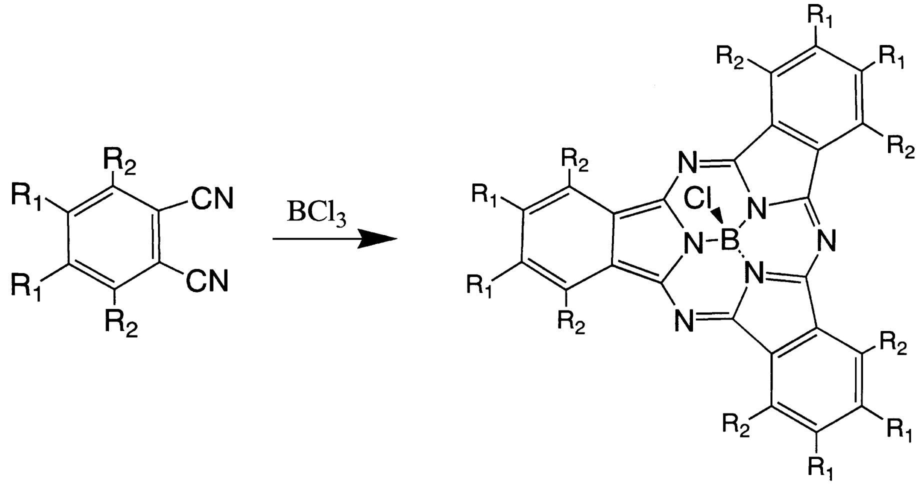
A solution of boron trichloride (20.5 ml, 0.02 mol, 1 M in hexane) was added into a mixture of 1,2-benzenedicarbonitrile (5.0 g, 0.04 mol) and 1-chloronaphthalene (12 ml) under argon atmosphere at −3 °C. After addition, the reaction mixture was heated to 120 °C while being stirred. Hexane was distilled off. It was heated to 250 °C for 10 min and then cooled to room temperature. The product was extracted with petroleum ether for 24 h and then toluene for 2 h on a Soxhlet extractor. The obtained product was recrystallized from ethanol (Scheme 1).

2.1.2. SubPC derivatives and their precursors

*2.1.2.1. 1,2-Dicyano-3,6-bis(thiobutyl)-4,5-difluorobenzene (**1**).* A mixture of 1,2-dicyano-3,4,5,6-tetrafluorobenzene (1.3 g, 6.53 mmol), butanethiol (1.18 g, 13.1 mmol), potassium fluoride (1.14 g, 19.6 mmol) and acetonitrile (7 ml) was heated at 50 °C for 24 h. After cooling, the organic layer was extracted with diethyl ether. The solution was washed with sodium hydroxide solution and saturated sodium chloride solution. The solvent was removed as a yellowish oil.

*2.1.2.2. 1,2-Dicyano-3,6-bis(thiophenyl)-4,5-difluorobenzene (**2**).* A mixture of 1,2-dicyano-3,4,5,6-tetrafluorobenzene (1.3 g, 6.53 mmol), thiophenol (1.44 g, 13.1 mmol), potassium fluoride (1.14 g, 19.6 mmol) and acetonitrile (7 ml) was heated at 50 °C for 2.5 h. The reaction product was washed with methanol and hot water, and filtered.

*2.1.2.3. 1,2-Dicyano-3,4,5,6-tetrakis(thiobutyl) benzene (**3**).* A mixture of 1,2-dicyano-3,4,5,6-tetrafluorobenzene (1.3 g, 6.53 mmol), butanethiol (2.93 g, 32 mmol), potassium fluoride (2.8 g, 48 mmol) and acetonitrile (10 ml) was heated at 50 °C for 144 h. After cooling, the organic layer was



Scheme 1. (1) SubPC: $R_1 = R_2 = H$, (2) SubPC: $R_1 = F$, $R_2 = S$-butyl, (3) SubPC: $R_1 = F$, $R_2 = S$-phenyl, (4) SubPC: $R_1 = R_2 = S$-butyl, (5) SubPC: $R_1 = R_2 = S$-phenyl, (6) SubPC: $R_1 =$ *tert*-butyl or H, $R_2 = H$, (7) SubPC: $R_1 = R_2 = F$.

extracted with diethyl ether. The solution was washed with sodium hydroxide solution and saturated sodium chloride solution. Solvent was removed.

*2.1.2.4. 1,2-Dicyano-3,4,5,6-tetrakis(thiophenyl) benzene (**4**).* A mixture of 1,2-dicyano-3,4,5,6-tetrafluorobenzene (1.3 g, 6.53 mmol), thiophenol (2.88 g, 26.1 mmol), potassium fluoride (2.28 g, 39.2 mmol) and acetonitrile (7 ml) was heated at 50 °C for 18 h. The reaction product was washed with methanol and hot water, and filtered.

2.1.2.5. SubPC derivatives. Each SubPC derivative was synthesized in accordance with the abovementioned SubPC synthetic method.

2.2. Spectroscopy

Absorption spectra were recorded on a Shimadzu UV-2400PC spectrophotometer. Fluorescence spectra were measured on a Hitachi F-4500 fluorescence spectrometer by using a solid sample holder, which was also used for a solution in order to minimize the effect of an internal light filter. Infrared (IR) spectra were recorded on a Shimadzu FTIR-8100A spectrometer. Proton nuclear magnetic resonance (^{1}H-NMR) spectra were measured at 400 MHz on a Bruker Avance 400S in chloroform-*d* ($CHCl_3$-*d*) using tetramethylsilane (TMS) as an internal standard.

2.3. Cyclic voltammetry

Cyclic voltammetry was carried out with a BAS CV-50W voltammetric analyzer at room temperature in acetonitrile containing a 0.01 mol dm^{-3} solution of tetrabutylammonium perchlorate (TBAP) [9–11]. Cyclic voltammograms were recorded by scanning the potential at the rate of 50 mV s^{-1}. The working and counter electrodes were platinum wires, and the reference electrode was a silver/silver chloride (Ag/AgCl) saturated sodium chloride electrode [12]. The area of the working electrode was 2.0×10^{-2} cm^2.

2.4. Laser flash photolysis

Laser flash photolysis in film was performed using a total reflection sapphire cell (10×30 mm, 1 mm thickness, and both side were cut at a 45° angle) [13,14], which was spin-coated with a thick of 1.2 μm SubPC containing 10% poly(methylmethacrylate) (PMMA) in cyclohexane photopolymer film as shown in Fig. 2. The excitation light pulse from a spectron laser system model SL402 YAG laser was expanded over the entire sample cell. A xenon lamp was used a monitoring beam [13,14].

The measurement was repeated five times within less than 3 wt.% of SubPC and 2,4,6-Tris(trichloromethyl)-1,3,5-triazine (TCT) as a quencher; more than 90% of the 355 nm-laser light absorbed in the sample film was absorbed by SubPC.

Films were prepared as follows; SubPC was added to this solution either by dissolving the dyes directly in polymer solution or by mixing a potion of concentrated dye solution. Films were adjusted to be 1.2 μm thick by spin-coating a solution onto a sample cell.

The photosensitivity of the photo initiator system was measured as follows; the sensitive layer which was prepared by coating a cyclohexane solution of the photosensitive composition containing 5 wt.% of the SubPC, 5 wt.% of TCT, 45 wt.% of trimethylrolpropane triacrylate and 45 wt.% of PMMA (90:10 mol%, MW = 50,000) onto a grained aluminium plate and exposed at 550 nm by the use of a xenon lamp, which was isolated using a monochrometer.

3. Results and discussion

3.1. SubPC and its derivatives

Scheme 1 shows the synthetic pathway of SubPC and its derivatives. SubPC was synthesized from 1,2-dicyanobenzene. SubPCF$_{12}$ was synthesized from 1,2-dicyano-3,4,5,6-tetrafluorobenzene. SubPC(*S*-butyl)$_6$F$_6$, SubPC(*S*-butyl)$_{12}$, SubPC (*S*-phenyl)$_6$F$_6$ and SubPC(*S*-phenyl)$_{12}$ were synthesized from precursors **1**–**4**, respectively.

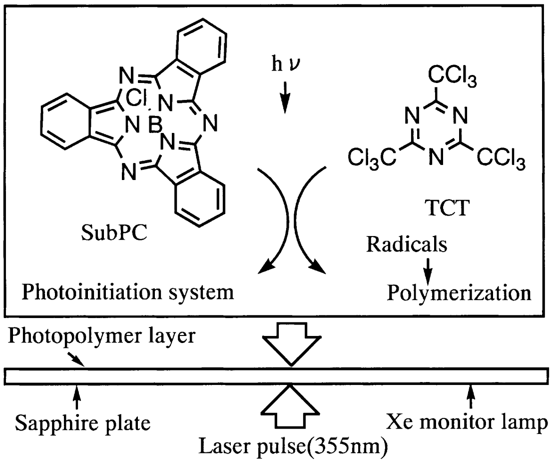


Fig. 2. Laser flash photolysis in film.

The precursors of SubPC derivatives were analyzed. The results are shown in Table 1. The analytical data were in good agreement with the proposed structures.

The target compounds, SubPC and its derivatives, were analyzed by UV, IR and ^{1}H-NMR spectra. The results are shown in Table 2.

The analytical data for SubPC and its derivatives were in good agreement with the proposed structure. Absorption bands in the IR spectrum of SubPC and its derivatives are assigned as follows: peaks around 1600 cm^{-1} are aromatic ring absorptions; in the region of 1200–1050 cm^{-1} are mainly absorption of out-of-plane bending of CH and NH in pyrrole rings, in which cyclotrimarization is found. These absorption bands in the IR spectrum are characteristic peaks of phthalocyanine derivatives.

Absorption maximum of SubPC and its derivatives appeared around 560–630 nm in the benzene solution. The strongest absorption peak assignees are the Q band which could be attributed to the allowed π–π* transition of the SubPC ring. The Q band absorption of SubPC and its derivatives shifted by around 100 nm to a shorter wavelength in comparison with usually metal phthalocyanine derivatives which appeared around 650 nm. The shift of absorption maxima depends upon the change in electron distribution in the size of macrocycle.

Absorption maxima of SubPC derivatives shifted to longer wavelength with increasing molecular weight.

The synthesized SubPC and its derivatives were soluble enough some organic solvents such as dichloromethane, chloroform, acetone and *N,N*-dimethylformamide (DMF).

3.2. Cyclic voltammogram

Cyclic voltammetry is used in estimations of electrochemistry. Cyclic voltammetry consists of cyclic potential of a stationary electrode immersed in a quiescent solution and measuring the resulting current. The excitation signal is a linear potential

Table 1
Analytical data of intermediates **1**–**4** of subphthalocyanine derivatives

Compound	Yield (%)	υ_{max} KBr/cm^{-1}	δ/ppm CHCl$_3$-*d*	Formula	MS[M]+ *m/z*	Found (calculation)		
						C	H	N
1	76	2950 (υ C-H), 2850 (υ C-H), 2250 (υ C-H), 1600 (υ C-C), 1580 (υ C-C), 1450 (υ C-C), 1375 (υ C-F), 1280 (υ C-F), 1230 (δ C-H), 825 (υ C-S)	0.89(*t*, 6H), 1.53(*m*, 8H), 3.15(*t*, 4H)	$C_{16}H_{18}N_2S_2F_2$	340	56.44 (56.44)	5.33 (5.33)	8.23 (8.23)
2	76	2980 (υ C-H), 2150 (υ C-N), 1600 (υ C-C), 1500 (υ C-C), 1450 (υ C-C), 870 (δ C-H)	0.98(*t*, 12H), 1.45(*m*,16H), 3.13(*t*, 8H)	$C_{24}H_{36}N_2S_4$	481	59.95 (59.95)	7.55 (7.55)	5.83 (5.83)
3	97	2250 (υ C-N), 1580 (υ C-C), 1520 (υ C-C), 1475 (υ C-C), 1450 (υ C-C), 1300 (υ C-F), 1080 (δ C-C), 750 (υ C-S)	7.3(*s*, 10H)	$C_{28}H_{10}N_2S_2F_2$	477	50.57 (50.57)	2.12 (2.12)	5.88 (5.88)
4	90	3050 (υ C-H), 2250 (υ C-N), 1600 (υ C-C), 1580 (υ C-C), 1480 (υ C-C), 1450 (υ C-C), 1300 (υ C-F), 750 (υ C-S)	7.25(*m*, 20H)	$C_{32}H_{20}N_2S_4$	561	68.53 (68.53)	3.59 (3.59)	4.99 (5.00)

scan with a triangular waveform. This triangular potential excitation signal sweeps the potential of the working electrode. The triangle returns at the same speed and permits the display of a complete voltammogram. Therefore, if a molecular is reduced in the forward scan, it will be reoxidized on the reverse scan. The current response shows the upper half, cathodic (reduction) and the lower half, an anodic (oxidation) peak.

Figs. 3 and 4 show cyclic voltammograms of SubPC and SubPC(*S*-phenyl)$_6$F$_6$. The reduction and oxidation potentials of SubPC and its derivatives are summarized in Table 3. The reported potentials are the midpoint potential of the anodic and cathodic peaks for each couple E_{mid}, and the peak potential for an irreversible step.

In general, oxidations of transition metal phthalocyanine derivatives are electrochemically irreversible, and the potential is about 1.0 V vs. standard hydrogen electrode (SHE) [15,16].

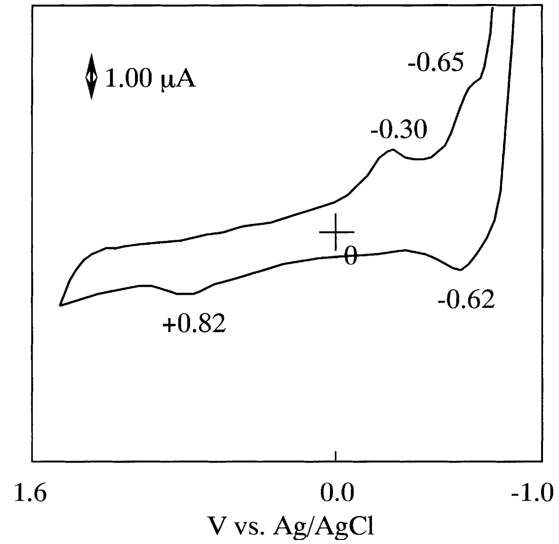


Fig. 3. Cyclic voltammogram of subphthalocyanine in acetonitrile with tetrabutylammonium perchlorate.

The cyclic voltammogram of SubPC showed two cathodic peaks at −0.30 and −0.65 V vs. Ag/AgCl, and two anodic peaks at 0.82 and −0.62 vs. Ag/AgCl. SubPC has two irreversible oxidation and reduction at 0.82 and −0.30 V vs. Ag/AgCl, and one pair of reversible reduction potential at −0.64 V vs. Ag/AgCl.

The reduction and oxidation behavior of M-PC derivatives are due to the interaction between the phthalocyanine ring and the central metal [17]. In

Table 2
Synthetic results of subphthalocyanine and its derivatives

Compound	Yield (%)	$\upsilon_{\mathbf{max}}$ KBr/cm^{-1}	δ/ppm CHCl$_3$-*d*	$\lambda_{\max}$/nm	Formula	MS[M]+ *m*/*z*	Found (calculation)		
							C	H	N
SubPC	71	3200 (υ C-H), 3070 (υ C-C), 2370 (υ C-N), 1611 (υ C-C), 1452 (υ C-C), 1304 (δ C-N), 1130 (δ C-H), 950 (υ C-S)	(*s*, 12H)	564	$C_{24}H_{12}N_6BCl$	431	66.88 (66.88)	2.81 (2.81)	19.50 (19.59)
Tert-butylSubPC	39	2980 (υ C-C), 1720 (υ C-C), 1620 (υ C-C), 1450 (δ C-H), 1400 (δ C-H), 1360 (δ C-H), 1280 (δ C-H), 1190 (δ C-H), 760 (δ C-H)	(*t*, 6H), (*m*, 32H), (*t*, 4H), (*s*, 2H)	566	$C_{36}H_{36}N_6BCl$	599	72.18 (72.18)	6.06 (6.06)	14.01 (14.01)
SubPCF$_{12}$	26	1490 (υ C-H), 1450 (υ C-N), 1270 (υ C-F), 1220 (υ C-F), 1120 (δ C-H), 960 (δ C-H), 800 (δ C-H), 760(δ C-H)	–	572	$C_{24}N_6BClF_{12}$	647	50.39 (50.39)	– (0.00)	14.70 (14.70)
SubPC(*S*-butyl)$_6$F$_6$	8	2980 (υ C-H), 2150 (υ C-N), 1600 (υ C-C), 1500 (υ C-C), 1450 (υ C-C), 1150 (δ C-H), 800 (δ C-H)	0.90 (*t*, 18H), 1.51(*m*, 24H)	596	$C_{48}H_{54}N_6BClF_6S_6$	1068	53.99 (53.99)	5.10 (5.10)	7.84 (7.87)
SubPC(*S*-phenyl)$_6$F$_6$	5	3050 (υ C-H), 2250 (υ C-N), 1580 (υ C-C), 1450 (υ C-C), 1250 (υ C-F), 1090 (δ C-H), 670 (δ C-H)	7.26 (*m*, 30H)	621	$C_{60}H_{30}N_6BClF_6S_6$	1188	60.66 (60.66)	2.55 (2.55)	7.08 (7.08)
SubPC(*S*-butyl)$_{12}$	17	2980 (υ C-H), 2125 (υ C-N), 1580 (υ C-C), 1500 (υ C-C), 1460 (υ C-C), 1000 (δ C-H), 800 (δ C-H)	0.91(*t*, 36H), 1.50 (*m*, 48H),	612	$C_{72}H_{108}N_6BClS_{12}$		58.07 (58.07)	7.31 (7.31)	5.65 (5.65)
SubPC(*S*-phenyl)$_{12}$	13	3080 (υ C-H), 2190 (υ C-N), 1600 (υ C-C), 1500 (υ C-C), 1460 (υ C-C), 1000 (δ C-H), 800 (δ C-H)	7.26 (*m*, 60H)	634	$C_{96}H_{60}N_6BClS_{12}$		66.67 (66.67)	3.50 (3.50)	4.86 (4.86)

the case of M-PC derivatives having cobalt as the central metal, phthalocyanine ring oxidation occurred after the central metal, and reduction was followed by the reduction of the central metal [15–17].

In the case of M-PC derivatives, the porphyrazine ring in the molecule is influenced by the π electrons around the closed system. The π electron system of M-PC derivatives consists of one porphyrazine and four phynylene rings [10,18,19]. Substituents of M-PC derivatives influence the π electron environment in the molecule especially the four phynylene rings. It is thought that the effect of substituents gives rise to the change of electron density of the four phynylene rings in the molecules of M-PC derivatives. Electron transfer properties of M-PC derivatives depend on the kind of the substituents.

While in the case of SubPC and its derivatives, redox potentials were had various values. However, one irreversible reduction potential certainly appeared around −0.3 V vs. Ag/AgCl.

It is concluded that the irreversible peaks around −0.3 V vs. Ag/AgCl are attributed to the reduction of the subphthalocyanine ring. The difference of cyclic voltammogram between SubPC derivatives

Table 3
Reduction and oxidation potential of subphthalocyanine and its derivatives

Compounds	Potential (V vs. Ag/AgCl)	
	Reduction	Oxidation
SubPC	−0.64−0.30[a]	0.82[a]
ΔE[b]	0.03	
tert-ButylSubPC	−0.51[a]	0.25[a] 0.79[a]
SubPC(*S*-butyl)$_6$F$_6$	−0.56[a] −0.28[a]	0.83
ΔE[b]		0.03
SubPC(*S*-phenyl)$_6$F$_6$	−0.27[a]	0.80[a] 1.44[a]
SubPC(*S*-butyl)$_{12}$	−0.61[a] −0.37[a]	0.18[a]
SubPC(*S*-phenyl)$_{12}$	−0.57[a] −0.36[a]	0.18[a]

Potentials of reversible wave are midpoint potential of anodic and cathodic peaks for each couple, E_{mid}.

[a] Irreversible peak.

[b] The anodic peak to cathodic neak seoaration for reversible couple.

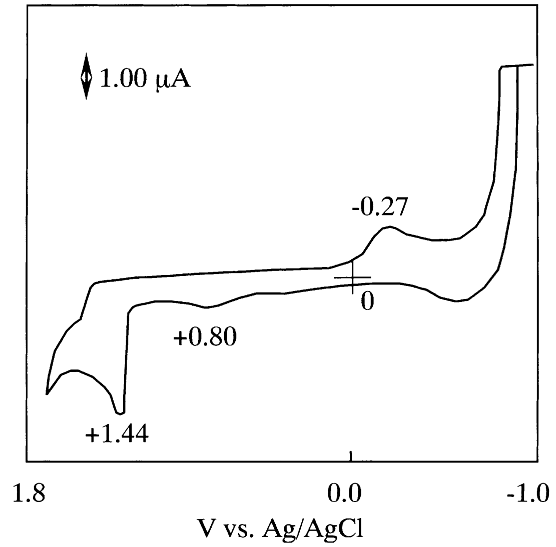


Fig. 4. Cyclic voltammogram of hexakis(*S*-phenyl)hexafyluoro-subphthalocyanine in acetonitrile with tetrabutylammonium perchlorate.

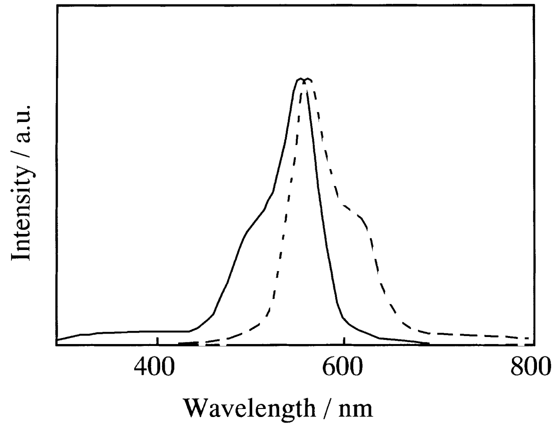


Fig. 5. Absorption and fluorescence spectra of subphthalocyanine. Absorption spectrum (—); fluorescence spectrum (- - - -).

is attributed to the variation of the substituents depends on the subphthalocyanine ring.

3.3. Laser flash photolysis

Fig. 5 shows the shapes of excitation and fluorescence spectra of SubPC. Excitation and fluorescence spectra of SubPC and its derivatives have almost the same profile (Table 4). No significant change on the fluorescence spectra were observed for SubPC and its derivatives in the fluorescence on an excitation at different wavelength, 355 and 420 nm.

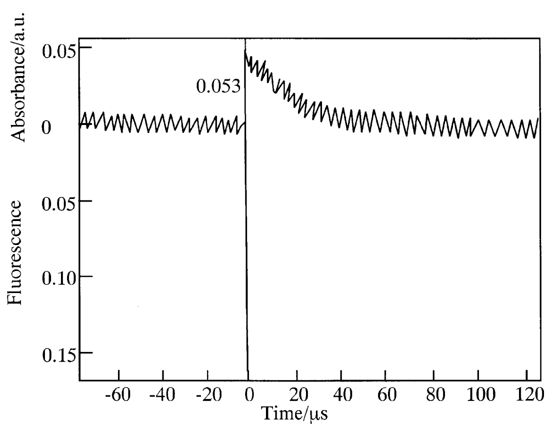
The fluorescence peak and transient decay were observed upon the excitation at a 355 nm pulse of a PMMA film containing SubPC. The photo-excited triplet state lifetime of SubPC was estimated as 20 μs laser pulse, as shown in Fig. 6. The transient absorption was assigned to a triplet state [20].

For the laser excitation of PMMA film at excitation of 550 nm using fluorescence spectrometer, the strongest fluorescence of SubPC was efficiently quenched by TCT. The fluorescence intensity was reduced with the concentration of TCT from 0 to 0.15 M. The fluorescence intensity is in direct proportion to the concentration of the TCT [21], the relationship gives a good straight line and it exhibits the Perrin type static quenching process [20]. The quenching radius (*Rf*) was determined from the slope of the logarithmic plot, and was estimated to be 15 nm.

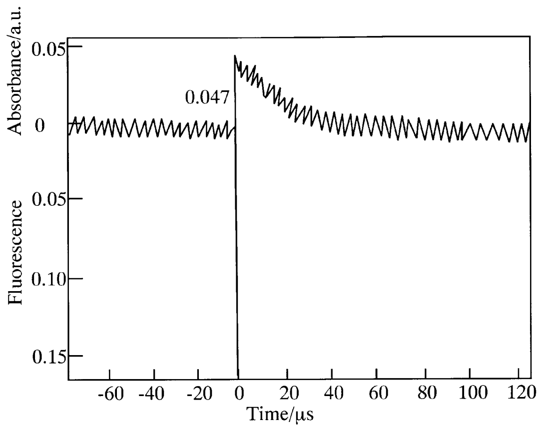
The triplet state of SubPC in a PMMA film was also quenched by TCT, the initial absorptions were decreased with the concentration of TCT, as shown in Fig. 7. In this time, the triplet life times measured almost same. The logarithmic plots of the initial absorption at various concentrations of

Table 4
Excitation and fluorescence maxima of subphthalocyanine derivatives in benzene solution

Compounds	Excitation (λ_{max}/mm)	Fluorescence (F_{max}/mm)
SubPC	564	569
tert-ButylSubPC	566	576
SubPCF$_{12}$	574	583
SubPC(*S*-phenyl)$_6$F$_6$	615	636
SubPC(*S*-phenyl)$_{12}$	632	667



In the absence of TCT



In the presence of 0.061 mol dm^{-3} of TCT

Fig. 6. Decay trace of subphthalocyanine on 450 nm. Substance concentration; 0.15 mol dm^{-3}. Excitation wavelength; 335 nm.

TCT were fitted to the Perrin equation, the triplet quenching radius (*Rt*) were calculation to be 1.3 nm, which was almost the same as *Rf*.

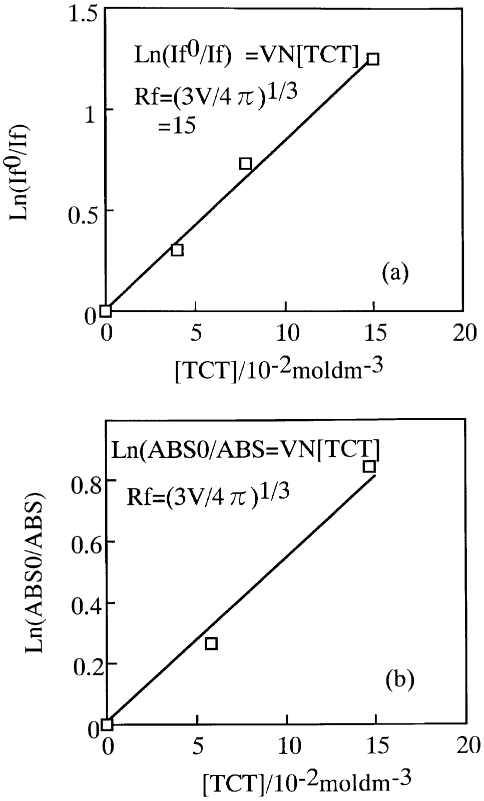


Fig. 7. Quenching of subphthalocyanine by 2,4,6-Tris(trichloromethyl)-1,3,5-triazine in poly(methylmethacrylate). (a) Fluorescence on 572 nm. Substance concentration; 0.03 mol dm^{-3}. Excitation wavelength; 550 nm. (b) Triplet absorption intensity on 450 nm. Substance concentration; 0.15 mol dm^{-3}. Excitation wavelength; 335 nm.

The results indicate the triplet initial absorption was not quenched by TCT, since the triplet state generally came from the fluorescent singlet state, and the triplet is apparently reduced with the decrease of the singlet state.

The redox potentials of SubPC were measured by cyclic voltammetry. Of SubPC, the reduction potentials showed at −0.64 V vs. Ag/AgCl and the oxidation potential appeared at 0.82 V vs. Ag/AgCl. It implies the possibility of the electron transfer from the singlet at excited state of SubPC to the grand state of TCT. SubPC derivatives have similar phenomena to SubPC.

In summary, it is suggested that the static-singlet-quenching process from the singlet photoexcited SubPC to TCT is predominant in the sensitization.

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